IS 8111 (1986): N, N-Dimethylaniline [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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(Reaffirmed 2000)

Indian Standard

SPECIFICATION FOR N,N-DIMETHYLANILINE

(First Revision)

UDC 667·281 : 547·551·3

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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September 1986

Indian Standard

SPECIFICATION FOR N,N-DIMETHYLANILINE

(First Revision)

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Indian Standard

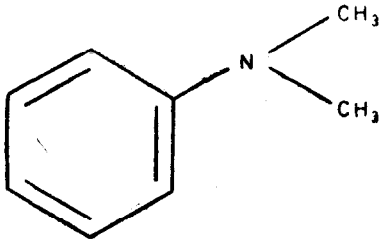
SPECIFICATION FOR N,N-DIMETHYLANILINE

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 26 February 1986, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 Dimethylaniline ($C_8H_{11}N$) is an important dye intermediate which finds extensive use in the manufacture of dyes, paper sensitizers, etc. It has the following structural formula:



N, N-Dimethylaniline
(Molecular mass 121)

0.3 This standard was first published in 1976. In view of the latest development in the dye intermediate industry and trade practices the Committee decided to revise the standard in order to cater to the need of manufacturer's and users of the material presently being made available in the country.

0.4 In the present version of the standard the requirement of freezing point has been revised and the method for assay has been modified. GLC method has been introduced for determination of monomethylaniline content.

0.5 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for N, N-dimethylaniline.

2. REQUIREMENTS

2.1 Description — The material shall be clear light yellow coloured liquid which tends to darken on storage.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR N, N-DIMETHYLANILINE

Sl No.	Characteristic	Requirement	Method of Test Ref to Cl No. in	
			Appendix A	IS : 5299-1969*
(1)	(2)	(3)	(4)	(5)
i)	Assay, percent by mass, *Min*	98·0	A-1	—
ii)	Relative density at 27°/27°C, *Max*	0·960	—	4
iii)	Freezing point, °C, *Min*	2·5	—	7
iv)	Distillation range, °C	95 percent shall distil between 191°C and 193°C	—	6
v)	Monomethylaniline content, percent by mass, *Max*	0·5	A-2	—

*Methods of sampling and test for dye intermediates.

3. PACKING AND MARKING

3 1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1979†) or as agreed to between the purchaser and the supplier.

*Rules for rounding off numerical values (*revised*).
†Specification for steel drums (galvanized and ungalvanized) (*second revision*).

3.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Gross mass, tare and net mass; and

d) Batch number.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in **3** of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for assay shall be conducted on each of the individual samples.

4.2.2 Tests for determination of remaining characteristics, namely, relative density, freezing point, distillation range and monomethylaniline content, shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirements of assay if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample, the test results for each of the characteristic shall satisfy the relevant requirement given in Table 1.

5. TEST METHODS

5.1 Test shall be carried out according to the methods prescribed in Appendix A and IS : 5299-1969*, as indicated in col 4 and 5 of Table 1.

*Methods of sampling and tests for dye intermediates.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

APPENDIX A

(*Clause* 5.1 and *Table* 1)

METHODS OF TEST FOR N, N-DIMETHYLANILINE

A-1. ASSAY

A-1.1 Reagents

A-1.1.1 *Acetic Acid*

A-1.1.2 *Acetic Anhydride*

A-1.1.3 *Methyl Violet Indicator* — Dissolve 30 mg of methyl violet in 100 ml of monochlorobenzene.

A-1.1.4 *Acetous Perchloric Acid* — 0·1 N. Mix 8·5 ml of 72 percent perchloric acid (AR grade) in 500 ml of glacial acetic acid in 1 000-ml volumetric flask. Add 20 ml of pure acetic anhydride (AR grade) and swirl the contents of the flask to ensure thorough mixing. Make up to the mark with glacial acetic acid and allow to stand overnight to ensure complete reaction of the acetic anhydride with the water present.

A-1.1.4.1 *Standardization of acetous perchloric acid* — Weigh accurately about 0·5 g potassium hydrogen phthalate (AR grade) in 100-ml beaker and add about 50 ml glacial acetic acid. Warm until dissolved and cool to room temperature. Add a few drops of methyl violet indicator and titrate the solution with perchloric acid. The colour changes from violet to blue. A very sharp end point is obtained by potentiometric titration using a glass and either a calomel or silver/silver chloride electrode.

NOTE 1 — Crystal violet indicator may also be used.

NOTE 2 — The acetous perchloric acid may also be standardized by 0·1 N sodium acetate solution with the same procedure as above.

A-1.2 Procedure — Weigh accurately 0·3 g of the material in a 250-ml beaker and dissolve in 50 ml of glacial acetic acid. Add 3 drops of methyl violet indicator. Run in the acetous perchloric acid from a burette, while stirring, until the colour changes from violet to green. A blank titration may be made with 50 ml glacial acetic acid in the presence of the indicator and a correction applied, if necessary.

*Specification for water for general laboratory use (*second revision*).

A-1.3 Calculation

$$\text{Assay, percent by mass} = \frac{V \times N \times M_1}{M_2 \times 10}$$

where

V = volume in ml of acetous perchloric acid used,

N = normality of acetous perchloric acid,

M_1 = molecular mass of amine, and

M_2 = mass in g of sample taken for the test.

A-2. DETERMINATION OF MONOMETHYLANILINE CONTENT

A-2.0 Outline of the Method — Monomethylaniline content in dimethylaniline is determined by gas liquid Chromatographic method. A sample of the material is injected into the gas chromatograph apparatus when it is carried by the carrier gas from one end of the column to the other end. During its movement the constituents of the sample undergo distribution at different rates and ultimately get separated from one another. The separated constituents emerge from the end of column one after the other and are detected by suitable means where response is related to the amount of a specific component leaving the column.

A-2.0.1 The chromatographic conditions given here are for guidance only. Variation in conditions to arrive at the typical chromatographic separation shown in Fig. 1 are possible.

A-2.1 Reagents

A-2.1.1 *Monomethylaniline* — Reagent grade.

A-2.1.2 *Aniline* — Reagent grade.

A-2.2 Apparatus — Any gas chromatograph capable of being operated under conditions suitable for resolving the individual constituents into distinct peaks may be used. A typical chromatogram obtained with Perkin Elmer Σ 3B instrument and the following conditions are shown in Fig. 1:

Column	Material:	glass or stainless steel
	Dimension:	2 000 mm length 2·75 mm internal dia
	Stationary phase:	15 percent Carbowax 20 M + 2 percent KOH on chromosorb GNAW. Mesh 80/100

Carrier gas:	nitrogen, flow rate, 30 ml/min	
Conditions:	injection temperature,	275°C
	detector temperature,	275°C
Detector:	flame ionization	
Chart Speed:	5 mm/min	
Sample:	0·4 microlitre	
Retention time:	dimethylaniline	5·23 minutes
	monomethylaniline	9·44 minutes
	aniline	10·67 minutes

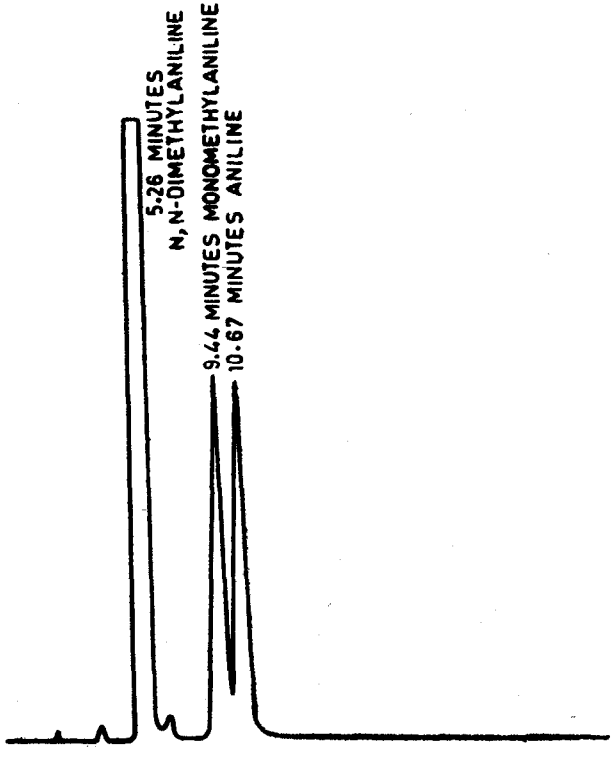


FIG. 1 CHROMATOGRAM FOR MONOMETHYLANILINE IN N,N-DIMETHYLANILINE

A-2.3 Procedure — Conduct the flow of the carrier gas and inject 0·4 ml of the sample at injection port where it is vaporized and well mixed with carrier gas. This is led into the chromatographic column wherein vaporized constituents of the sample are separated out by virtue of their differing interaction with the stationary phase. For the separation to be efficient it is necessary that the column is maintaned at the temperature suggested throughout the time required for the resolution of the constituents. As the sample enters the detector, it gives a signal corresponding to the amount of particular constituent leaving the column. The detector signal on transmission to the recorder, plots the chart. Run the chromatograph on synthetic mixture of dimethylaniline monomethylaniline and aniline also. From the specific area under various peaks corresponding to specific constituents, the quantities of different constituents are determined.

A-2.4 Calculation

A-2.4.1 Calculate the peak areas of individual constituent pertaining to monomethylaniline on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure monomethylaniline.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	Unit	Symbol
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	Unit	Symbol
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	Unit	Symbol	Definition
Force	newton	N	1 N = 1 $kg.m/s^2$
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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[*Page* 4, *clause* **2.2**, *Table* 1, *Sl No.* (iii), *col* 3] — Substitute '1.8' *for* '2.5'.

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Reprography Unit, BIS, New Delhi, India

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TO
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[*Page* 4, *Table* 1, *Sl No.* (ii) *Relative Density*] — Delete.

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Reprography Unit, BIS, New Delhi, India